Johns-Manville Research Center

Report No. 400-T-13 CONFIDENTIAL

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Title: DETERMINATION OF FREE SILICA IN VARIOUS JOHNS-MANVILLE PRODUCTS

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SUMMARY

Values for free silica in several Johns-Manville products have been determined by the X-ray diffraction method. Care was taken to select representative samples and to prepare representative portions of these for the analyses. As a check on the accuracy of the method, free silica values in two of the samples were determined by a chemical method. The results obtained show that the two methods agree within reasonable limits.

A value of 13 per cent quartz in Transite pipe fabricated from a mixture which originally contained 27.1 per cent ground silica (quartz) indicates that approximately half of the quartz has reacted.

Of the Zelienople fire bricks examined, JM-20, JM-23, and JM-26 contain moderate amounts of quartz, while JM-28 and JM-3000 contain practically no quartz. All the bricks examined contain some cristobalite, and in Nos. JM-23, JM-26 and JM-28 the cristobalite content is 30 per cent or greater.

Contents: Summary, Discussion, Results, and Comments.

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DISCUSSION

Samples Submitted

1. Transite pipe: 1-ft length of 6 in. Class 150 pressure pipe, made at Manville 10/27/58. Cut from pipe No. 381, made on machine No. 2; air cured for 16 hr, steam cured for 16 hr at 100 psi. As stated in letter of R. J. Gorman to A. B. Cummins, dated 10/31/58, this sample contains:

Fiber blend M558-24E	27.5%
Pioneer National Cement	45.4
Manville plant ground silica	27.1

2. Sample of rigid veneered Transite shingle, surf green Colorbestos siding. Supplied by E. R. Ihne.

3. Five samples of aluminum silicate insulating fire brick, obtained from the Zelienople plant by S. E. Wooley.

JM-20 brick
JM-23
JM-26
JM-28
JM-3000

Methods

Sampling. Drillings were made through a number of scattered areas of the Transite pipe wall, using a 1/2 in. Carbaloy drill, and boring entirely through the pipe wall each time. The resulting powder was collected in a 4-oz jar and mixed overnight on a set of rollers.

The Transite shingle was sampled in the same manner, each hole being drilled entirely through the shingle, so as to obtain proportionate amounts of the veneer and the interior.

Representative samples of the Zelienople bricks were obtained as follows. The entire brick was first crushed in a jaw crusher, then transferred to a 5-lb jar and mixed on the rollers and later by quartering. A portion of this (approximately 50 to 100 grams) was then ground to pass a 60 mesh screen, transferred to a 4-oz jar and mixed by rolling.

Preparation of Standards for X-ray diffraction Determinations. A set of quartz-plus-Transite standards was prepared, using a highly pure ground quartz obtained from the spectroscopy laboratory. For each of the following, 2-gram mixtures were prepared:

Transite	95 parts,	quartz	5 parts
"	90 "	"	10 "
"	80 "	"	20 "
"	70 "	"	30 "

Each mixture was finely ground by hand in a porcelain mortar and then transferred to a 9 ml specimen vial. Further mixing was then accomplished by overnight tumbling of the vials. Because of the fibrous components occurring in Transite it was found impracticable to grind these samples to pass a 325 mesh screen. However, grinding was continued until all of the particulate material had been reduced to a size range comparable to that of 325 mesh material.

A set of quartz-plus-brick standards was prepared, using the 60 mesh JM-28 brick, and following the method outlined above for Transite. Each mixture was ground to pass 325 mesh, and mixed further by tumbling.

Brick	95	parts	quartz	5	parts
"	90	"	"	10	"
"	85	"	"	15	"
"	80	"	"	20	"

Since preliminary X-ray diffraction diagrams showed cristobalite as well as quartz in these brick samples it was necessary to obtain some essentially pure cristobalite before any quantitative estimates could be made. A c.p. silicic acid which contained 0.005 per cent Cl as the only reported impurity was selected. Several grams of this were placed in a flat platinum dish provided with a platinum cover. This container was then placed in a gas-fired pot furnace, and a temperature of 1540C ±5 was maintained for several hours. The container was removed from the furnace while still glowing and immediately cooled in a jet of air. Subsequent petrographic examination by H. Insley and D. Bailey showed a cristobalite content of at least 98 per cent.

A set of cristobalite-plus-brick standards was prepared, using the above cristobalite, plus 60 mesh JM-20 brick. Each mixture was ground to pass 325 mesh, and mixed further by tumbling.

Brick	95	parts	cristobalite	5	parts
"	90	"	"	10	"
"	85	"	"	15	"
"	75	"	"	25	"
"	50	"	"	50	"

Determination of Free Silica in Transite Pipe and Transite Shingle by Chemical Method. Portions of the Transite pipe and the Transite shingle were submitted to the Analytical Section for determination of free silica. The method used was outlined in Report No. 492-Tr-2 of May 1948, and consists of treating the powdered sample first with dilute HCl, then with a dilute alkali solution. The resulting residues were then screened through a 100 mesh sieve, which separates quartz from crocidolite fiber and the fractions were examined petrographically by D. Bailey to determine the amounts of quartz present. The results are given by C. L. Williams in Report No. 492-Int-67, and are included in this report also.

Microscopic Examinations. All of the submitted samples were examined in powder form under the petrographic microscope. This method serves as a means of obtaining semi-quantitative estimates of the quartz content, but is of no value in determining the cristobalite content of fire brick samples, owing to the sub-microscopic character of cristobalite in these materials. Some attempt was made to observe microscopically whether any amorphous free silica occurs in any of the samples submitted.

X-ray Diffraction Procedures. X-ray diffraction patterns were obtained with a Norelco Diffractometer using nickel filtered copper Kα radiation.

All samples were scanned at 2° per minute from 3° to 90°2θ for qualitative analysis. From these diffractometer diagrams it was determined that:

a) these samples contain no measurable amount of tridymite;
b) all brick samples contain mullite;
c) JM-20 brick contains less cristobalite than any other brick;
d) JM-28 brick contains less quartz than any other brick.

JM-20 and JM-28 brick were selected for standard additions of cristobalite and quartz, respectively, preparatory to plotting standard curves.

For quantitative analysis samples were scanned in triplicate at ¼° per minute over the following lines:

3.34Å line for quartz in pipe and shingle samples;
4.25Å line for quartz in brick samples;
4.04Å line for cristobalite in brick samples.

The presence of mullite (major line 3.33Å) in all brick samples made it necessary to use the secondary (4.25Å) line for quartz determination in the brick samples. The use of this weak line for determining small amounts of quartz reduces the accuracy of the analysis.

Standard curves were prepared from a plot of the area under the diffractometer trace versus the known amount of quartz (or cristobalite) in the sample.

The unknown samples were scanned over the chosen line and the area under the diffractometer trace computed. Using this area, the quantitative reading was made directly from the appropriate standard curve.

RESULTS

Except as noted, the following values for free silica were determined by the X-ray diffraction method outlined in this report. The values for quartz in fire brick are correct to within ±10 per cent of the amounts reported. Values for cristobalite in fire brick and for quartz in Transite are correct to within ±5 per cent of the values reported. No tridymite was seen in any of the samples.

	Transite Pipe		Transite Shingle		Fire Brick Samples JM-20	JM-23	JM-26	JM-28	JM-3000
Quartz	14%*	13%	18%*	20%	8%	20%	5%	Trace**	Trace**
Cristobalite	-	-	-	-	5	38	45	30%	16%
Amorphous silica***	Faint trace				None seen				

*Determined by chemical method, as in Reports No. 492-Tr-2; 492-Int-67.
**Petrographic determinations only.
***Low refractive index (< 1.48); isotropic.